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**GB 0787187 A EP 0359068 A2 EP 0342011 A1
US 4828619 A**

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Online : CHEMENG, PATENTS****(54) Aeration admixture for cementitious compositions**

(57) An aeration admixture especially suitable for use with cementitious compositions comprising fly ashes with high levels of residual carbon (loss on ignition in excess of 5%) comprises a fatty acid-based surface active agent; and a non-ionic surface active agent, the fatty acid-based surface active agent (a) being selected from C₁₂₋₂₄ alkanoic acids and their alkali metal, lower alkylamine and lower alkanolamine salts, and the non-ionic surface active agent (b) being selected from materials of the formula



where Ph(R) represents a phenyl group substituted with R, R being C₈₋₉alkyl and n is from 1 - 50. An additional useful component is a salt selected from the group consisting of salts of alkyl sulphonates, alkylaryl sulphonates, sulphate esters of higher alcohols and resins.

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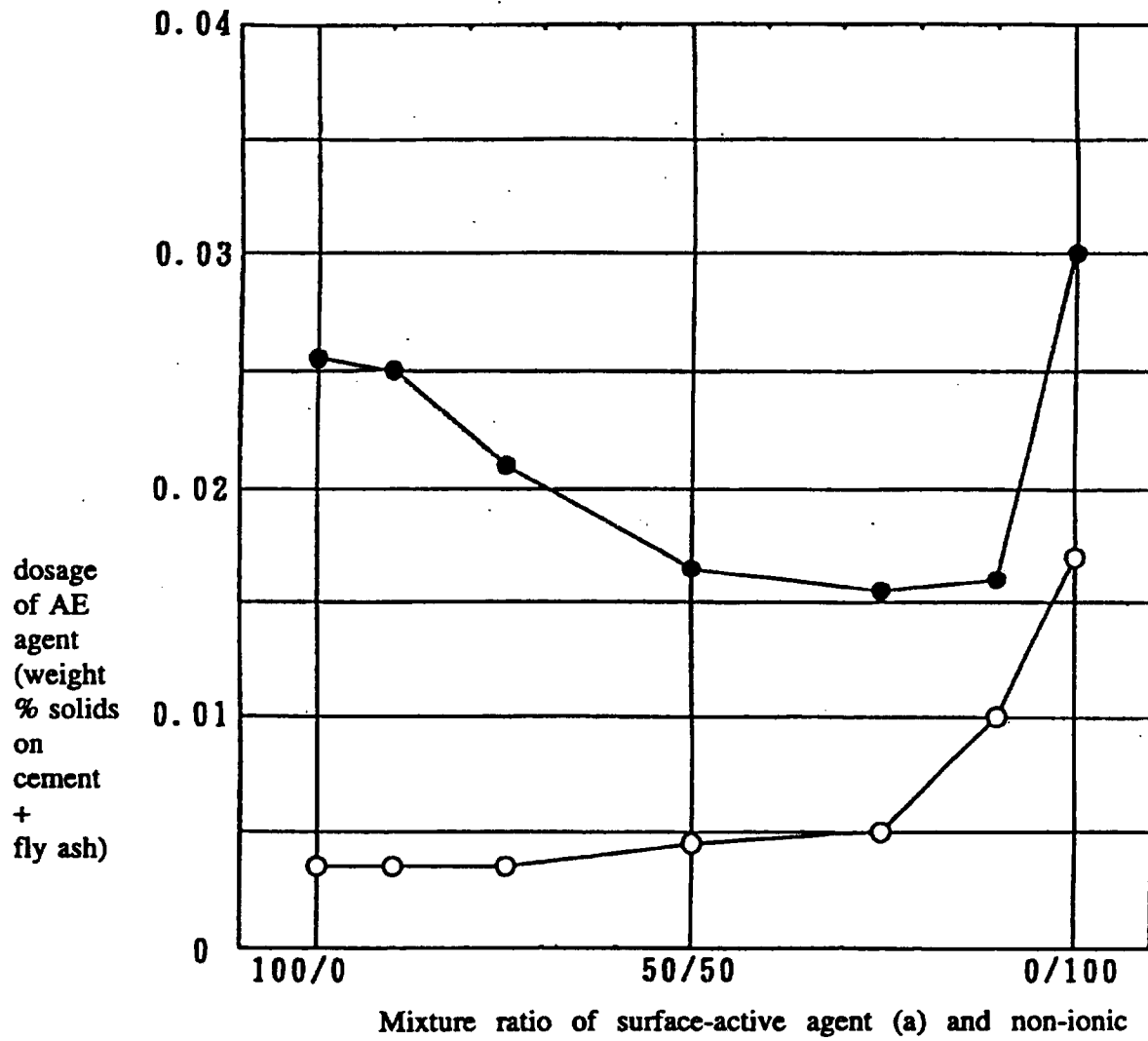


Fig. 1

Aeration Admixture

This invention relates to the entrainment of air in cementitious compositions and to air-entraining compositions for use therein.

Cementitious compositions such as concrete, mortar and grout sometimes need to be aerated, for example, to improve workability or to confer enhanced freeze-thaw durability.

- 5 This is commonly done by incorporating into the fluid composition an air-entraining admixture (hereinafter "AE admixture"). The act of mixing the cementitious composition causes bubbles to form; these are stabilised by the AE admixture. The materials themselves are described in ASTM C 260 and the subject of aeration is extensively covered in the literature (see, for example, "Concrete Admixtures Handbook", ed. Ramachandran (Noyes
10 1984) the disclosures of which are incorporated herein by reference). Examples of AE admixtures include surfactants, sulphate esters of higher alcohols and alkyl sulphonates.

- While the known AE admixtures have given excellent performance with many of the known cementitious compositions, they are not universally effective. One example of diminished effectiveness is use in conjunction with cementitious compositions which
15 comprise fly ash. Fly ash is a residue of industrial powdered coal-burning furnaces and is widely used in cementitious compositions, for example, as a permeability reducer. The problem with fly ashes is that they contain proportions of residual carbon (sometimes referred to as "uncombusted carbon") which survived the combustion process. It is believed that such residual carbon has the ability to absorb AE admixtures and thereby diminish
20 their effectiveness. The problem is compounded by two further factors, (i) coal may be sourced from a wide variety of areas, meaning that the residual carbon content will change from batch to batch, making it difficult to counteract the problem; and (ii) environmental pressures have forced the burning of coal at lower temperatures, thereby leaving even more residual carbon in the resulting fly ash.

- 25 Attempts have been made to overcome this problem by developing particular AE agents. However, these AE admixtures have either had poor air-entraining properties or have needed a large dose to achieve their effect, or both of these problems.

It has now been found that a particular blend of materials gives rise to an AE admixture which not only has excellent air-entraining properties but which also achieves these at relatively low doses, even in the presence of fly ash with residual carbon. There is therefore provided, according to the present invention, an air-entraining admixture which
5 comprises

- (a) a fatty acid-based surface active agent; and
- (b) a non-ionic surface active agent;

the fatty acid-based surface active agent (a) being selected from C_{12-24} alkanolic acids and their alkali metal, lower alkylamine and lower alkanolamine salts, and the non-ionic surface
10 active agent (b) being selected from materials of the formula



where Ph(R) represents a phenyl group substituted with R, R being C_{8-9} alkyl and n is from 1 - 50.

In a preferred embodiment of the invention, the air-entraining admixture additionally
15 comprises (c) a salt selected from the group consisting of the salts of alkyl sulphonates, alkylaryl sulphonates, sulphate esters of higher alcohols and resinsates.

The fatty acid-based surface active agent (a) may be selected from any such substance known to the art. The fatty acid chain in the surface-active agent (a) may be saturated or unsaturated, straight chain or branched chain. The fatty acid-based surface active agents (a)
20 may be fatty acids, or preferably, they may be salts of such fatty acids, preferably the salts of alkali metals or amines. The preferred alkali metal salts are those of sodium and potassium, and the preferred salts of amines are preferably those of low molecular weight alkylamines and alkanolamines, preferably those of triethylamine or triethanolamine. Preferred surface-active agents (a) include tall oil fatty acid soap, oleic acid soap, linoleic
25 acid soap and palm fatty acid soap, tall oil fatty acid soap being especially desirable.

The non-ionic surface active agent (b) as hereinabove defined may be selected from any such material. The substituent R on the phenyl group may be straight chain or branched chain, and is preferably C₈ or C₉ alkyl. Specific examples include polyoxyethylene nonylphenyl ether and polyoxyethylene octyl phenyl ether. The number n of oxyethylene units per molecule is in the range of 1 to 50. It has been found that the value of n has an effect on the ability of the AE admixtures of this invention to cause sufficient air-entraining in the presence of fly ash with a high proportion of residual carbon. For use with such a material, the AE admixture preferably comprises a surface-active agent (b) with from 20 - 30 oxyethylene units per molecule.

- 10 The salts of component (c) of the AE admixture according to the invention may be selected from a wide variety of suitable materials.

The alkyl radicals of alkyl sulphonate and alkyl aryl sulphonate are typically C₉-C₁₂ and may be straight chain or branched chain. The salts are preferably alkali metal salts, especially sodium and potassium, or triethanolamine salts. Specific types include α-olefin sulfonates, alkyl benzene sulphonates and alkyl sulphates. With regard to the high alcohol sulphates, the alcohols should have at least 12 carbon atoms. Ethylene oxide adducts of such alcohols are also useful, and typical examples include polyoxyethylene alkyl ether sulphates and polyoxyethylene alkyl phenyl ether sulphates. Typical resinsates include rosin soap obtained by saponification of pine resin with sodium hydroxide or potassium hydroxide, and sodium, potassium, triethanolamine salts of abietic acid.

The weight ratios of the individual components are from 10 - 90%, preferably from 10 - 80% (based on active material) of surface-active agent (a), from 90 - 10% from non-ionic surface-active agent (b) and no more than 20% of component (c) when this material is present. The admixture is used at a rate of from 0.001 to 0.1% by weight actives by weight of cement plus AE admixture. The AE admixtures are generally used in the form of aqueous solutions. They may be used in conjunction with other art-recognised admixtures such as water-reducing agents, AE water-reducing agents, high-range water-reducing agents, high-range AE water-reducing agents, fluidifiers, waterproofing agents, rust-inhibiting agents and shrinkage-reducing agents.

The AE admixture according to the invention is useful for the aeration of cementitious compositions. It is especially useful when used in such compositions which comprise materials comprising residual carbon. It is believed, without limiting the invention in any way, that the residual carbon absorbs conventional AE admixtures. The problems caused by materials such as fly ash are recognised in JIS (Japanese Industrial Standard) A 6201 which specifies that fly ashes of more than 5% loss on ignition should not be used. However, the AE admixtures of the present invention can be used with fly ashes with loss on ignition in excess of 5%. The invention therefore provides a process of preparing an aerated, fly ash-containing cementitious composition using a fly ash which has a loss on ignition of more than 5%, comprising the addition to the cementitious composition including the fly ash of an air-entraining admixture as hereinabove described. The invention further provides an aerated fly ash-containing cementitious composition in which the fly ash has a loss on ignition of more than 5%, the composition comprising an air-entraining admixture as hereinabove described.

The invention is further illustrated by the following examples.

Example 1

1) Method of Testing

a. Method of Mixing Concrete

Fine aggregate, cement, and mixing water (including AE water-reducing agent and AE admixture according to the invention) is introduced into a mixer and mixing is performed for 30 seconds. Coarse aggregate is then introduced and mixing is performed for 90 seconds.

b. Time-dependent Change in Concrete

After measuring slump (JIS A 1101) and air content (JIS A 1128) of the concrete mix, the mix is transferred to a tilting-type mixer, and agitation is carried out at an

angle of tilting of the mixer of 15 degrees and rotating speed of the mixer of 2 rpm, and air contents at 30 minutes and 60 minutes are measured.

2) Materials Used

a. Cement

5 Ordinary portland cement (specific gravity = 3.16) consisting of equal parts of ordinary portland cements of the Onoda, Sumitomo, and Mitsubishi Material companies are mixed together and used.

b. Fly Ash

10 Commercial fly ash (specific gravity = 2.26, specific surface = $3410 \text{ cm}^2/\text{g}$, loss on ignition = 3.9%, methylene blue absorption = 0.9 mg/g) is used.

c. Fine Aggregate

Oi River System pit sand (specific gravity = 2.64, fineness modulus = 2.76) is used.

d. Coarse Aggregate

15 Crushed stone from Ome, Tokyo (maximum size = 20 mm, specific gravity = 2.65, fineness modulus = 6.63) is used.

e. Mixing Water

Tap water is used.

f. AE Water-reducing Agent

An AE water-reducing agent (proprietary name: "Pozzolith" (trade mark) No. 70)

manufactured by NMB Ltd. is used.

g. AE Admixture

The following AE admixtures are used.

As fatty acid-based surface active agent (a):

- 5 - potassium hydroxide-saponified tall oil

As nonionic surface-active agent (b):

- the 10-, 20-, 25-, 30-, 40-, and 50-mol ethylene oxide adducts of polyoxyethylene nonylphenyl ether (hereinafter abbreviated to b1, b2, b3, b4, b5 and b6, respectively).

10 As ingredient (c):

- Dodecylbenzene sodium sulfonate (hereinafter abbreviated to c1)
- Potassium hydroxide-saponified resin acid (hereinafter abbreviated to c2)
- α -olefin sodium sulfonate (hereinafter abbreviated to c3)
- Polyoxyethylene nonylphenyl ether sodium sulfate (hereinafter abbreviated to c4)

15

3) Concrete Mix Proportions and Test Results

Concrete mix proportions are determined with target slump of 18 ± 2 cm and target air content of 5 ± 0.5 percent, without adding fly ash and with fly ash added as 20 percent of total cement plus fly ash. The mix proportions are given in Table 1.

Table 1

5	Fly Ash Content Rate FA/(C+FA) (%)	Water- Binder Ratio W/(C+FA)	Sand- Agg. Ratio s/a (%)	Unit	Content (kg/m ³)				
					W	C	FA	S	G AEWRA
	0	0.575	46	184	320	-	798	352	800 ml
	20	0.563	45	130	250	64	776	963	800 ml

W: water, C: cement, FA: fly ash, S: sand (fine aggregate) G: gravel (coarse aggregate),

10 AEWRA: air-entraining, water-reducing agent

Concrete is manufactured according to these mix proportions, and the air contents and time-dependent changes in air contents are tested. The results are given in Tables 2 and 3.

Table 2

Experi- ment No.	Fly Ash Content Rate %	AE Admixture Ingredient				(c) Kind	Rate	Dosage ¹⁾ (%)	Dosage Increment Rate ²⁾ %	Time-dependent Air Content Change (%)		
		(a) Rate	(b) Kind	(b) Rate	(b) Kind					0 min.	30 min.	60 min.
Comparison Example	1	0										
	2	0	100	b3	0	None	0	0.0035	-	4.9	5.1	4.8
	3	0	90	b3	10	"	0	0.0035	-	4.8	4.7	4.6
	4	0	75	b3	25	"	0	0.0035	-	5.1	4.9	4.6
	5	0	50	b3	50	"	0	0.0045	-	5.1	4.9	4.8
	6	0	25	b3	75	"	0	0.0050	-	5.2	4.8	4.3
	7	0	10	b3	90	"	0	0.0100	-	5.1	4.8	4.4
	8	0	0	b3	100	"	0	0.0170	-	5.2	4.5	4.2
	9	20	0	b3	0	c2	100	0.0006	-	5.1	4.7	4.4
	10	20	100	b3	0	c2	100	0.0040	667	5.0	3.0	2.4
Example	11	20	90	b3	10	None	0	0.0250	714	5.3	5.0	4.8
	12	20	75	b3	25	"	0	0.0210	600	5.4	5.2	4.9
	13	20	50	b3	50	"	0	0.0165	367	5.0	4.8	4.6
	14	20	25	b3	75	"	0	0.0155	310	5.2	4.7	4.6
	15	20	10	b3	90	"	0	0.0160	160	5.4	5.1	4.6
Comparison Example	16	20	0	b3	100	None	0	0.0300	143	5.1	4.6	3.6
Example	17	20	50	b1	50	None	0	0.0100	-	5.1	4.9	4.4
	18	20	50	b2	50	"	0	0.0145	-	4.9	4.7	4.5
	19	20	50	b4	50	"	0	0.0200	-	5.2	4.8	4.6
	20	20	50	b5	50	"	0	0.0240	-	5.0	4.7	4.5
	21	20	50	b6	50	"	0	0.0240	-	5.0	4.8	4.6

Note ¹⁾ Dosage of AE admixture solids percent by weight of cement or total quantity of cement and fly ash.

²⁾ Rate of AE admixture dosage when using fly ash with dosage when not using fly ash as 100 percent.

Experiments No. 13 and Nos. 17 to 21 in Table 2 are cases where the number of oxyethylene units added to polyoxyethylene nonylphenyl ether is varied, being 10 (b1), 20 (b2), 25 (b3), 30 (b4), 40 (b5) and 50 (b6), surface-active agent (a) being held constant. A tendency is seen for the dosage of AE admixture to decrease as the number of oxyethylene units is decreased. Further, as the solubility of nonionic surface-active agent (b) decreases with decreasing number of oxyethylene units, for practical purposes it is desirable for the number of oxyethylene units added to be in the range of 20 to 30.

Experiments No. 1 to No. 7 are cases where no fly ash has been added, and when the addition rate of ingredient (b) is increased, a tendency is seen for the dosage of AE admixture needed for attaining the target air content to be higher compared with the case where there is no addition of the ingredient (b) (Experiment No. 1). However, in the cases of Experiments No. 10 to No. 16 in which fly ash is used, the dosage of AE admixture needed to attain the target air content, when the addition rates of the ingredient (b) are from 10 to 90 percent, are lower compared with those needed in the cases of surface-active (a) used alone (Experiment No. 10) and nonionic surface-active agent (b) used alone (Experiment No. 16). The smallest dosage is obtained when the ratio of the ingredients (a) and (b) is 25 : 75 weight percent. This can be seen with reference to Figure 1, a graph of dosage versus composition.

Experiments No. 8 and No 9 are cases in which a commercial resinate type AE admixture (c2) is used, and although the target air content is obtained immediately after mixing, in the case of the addition of fly ash (Experiment No. 9) air content is greatly reduced with elapse of time.

Table 3 gives the results when dodecylbenzene sodium sulfonate (c1), potassium hydroxide-saponified resin acid (c2), α -olefin sodium sulfonate (c3) and polyoxyethylene nonylphenylether sodium sulphate (c4) are used in combination added as (c) to the ingredients (a) and (b). Experiments No. 26 to No. 31 in Table 3 are cases where fly ash is added, and the AE admixtures using these ingredients exhibit excellent properties at small dosages. Experiment No. 32 is a case where potassium hydroxide-saponified resin acid is used at the higher-than-desirable mixing ratio of 25 weight percent, and air content decreases with elapse of time.

Table 3

Experiment No.	Fly Ash Content Rate %	AE Admixture Ingredient				Dosage ¹⁾	Dosage Increment Rate ²⁾ %	Time-dependent Air Content Change (%)		
		(a) Rate	(b) Kind	(b) Rate	(c) Kind	(%) Rate		0 min.	30 min.	60 min.
Comparison Example	22	0	40	b3	50	c1	10	0.0040	-	5.0 4.8 4.7
	23	0	40	b3	50	c2	10	0.0040	-	4.8 4.6 4.4
	24	0	40	b3	50	c3	10	0.0035	-	4.9 4.7 4.5
	25	0	40	b3	50	c4	10	0.0040	-	4.6 4.4 4.2
Example	26	20	40	b3	50	c1	10	0.0140	350	5.1 5.0 4.7
	27	20	35	b3	50	c2	5	0.0155	-	4.8 4.6 4.4
	28	20	40	b3	50	c2	10	0.0145	413	4.9 4.8 4.6
	29	20	45	b3	50	c2	15	0.0135	-	4.7 4.5 4.4
	30	20	40	b3	50	c3	10	0.0140	414	4.9 4.7 4.5
	31	20	40	b3	50	c4	10	0.0145	362	5.2 5.0 4.8
Comparison Example	32	20	25	b3	50	c2	25	0.0100	-	5.0 4.0 3.1

Note ¹⁾ Dosage of AE admixture solids percent by weight of cement or total quantity of cement and fly ash.

²⁾ Rate of AE admixture dosage when using fly ash with dosage when not using fly ash as 100 percent.

Example 21) **Method of Testing**a. **Method of Mixing Concrete - as per Example 1**

Slumps (JIS A 1101) and air contents (JIS a 1128) of concretes are measured.

5 2) **Materials Used**a. **Cement**

Ordinary portland cement (specific gravity = 3.16) consisting of equal parts of ordinary portland cements of the Onoda, Sumitomo and Mitsubishi Material companies mixed together is used.

10 b. **Fly ash**

Eight lots (F1 to F8) of fly ashes of different losses on ignition produced from the same power station are used. The specific gravities, losses on ignition and methylene blue absorptions of these fly ashes are given in Table 5.

c. **Fine Aggregate**

15 Oi River System pit sand (specific gravity = 2.64, fineness modules = 2.76) is used.

d. **Coarse Aggregate**

Crushed stone from Ome, Tokyo (maximum size = 20 mm, specific gravity = 2.65, fineness modules = 6.63) is used.

e. **Mixing Water**

20 Tap water is used.

f. **AE admixture**

The AE admixtures described hereinunder are used.

- **AE1**

25 Potassium hydroxide-saponified tall oil as fatty acid base surface active agent (a) in a proportion of 50 weight percent and a polyoxyethylene nonylphenyl ether adduct having 25 oxyethylene units as nonionic surface active agent (b) in a proportion of 50 weight percent are mixed together.

- AE2

5 Potassium hydroxide-saponified tall oil as fatty acid-based surface agent (a) in a proportion of 35 weight percent, polyoxyethylene nonylphenyl ether adduct having 25 oxyethylene units as nonionic surface active agent (b) in a proportion of 60 weight percent, and potassium hydroxide-saponified resin acid in a proportion of 5 weight percent are mixed together.

- AE3

An AE admixture manufactured by NMB Ltd. (proprietary name: No. 303A, with an alkylaryl sulfonate as the main ingredient).

10 - AE4

An AE admixture for fly ash of Toho Kagaku Kogyo (proprietary name: "Cemerol" T-80, with polyoxyethylene sorbitan mono-oleate as the main ingredient)

3) Concrete Mix Proportions and Test Results

15 The dosage of AE admixture which gave an air content of approximately 5 percent when using fly ash F4 of roughly median loss on ignition is determined. This dosage is used for the other fly ashes and the fluctuations in air content are measured.

20 The mix proportions when fly ash is used at 20 percent by total weight of cement plus fly ash are determined by trial mixes. The mix proportions are shown in Table 4.

Table 4

Fly Ash Content Rate FA/(C+FA) (%)	Water- Binder Ratio W/(C+FA)	Sand- Agg. Ratio s/a (%)	Unit Content (kg/m ³)				
			W	C	FA	S	G
20	0.60	45	180	240	60	788	981

W: water, C: cement, FA: fly ash, S: sand (fine aggregate) G: gravel (coarse aggregate)

The test results are given in Tables 5 and 6. As shown in Table 5, whereas air contents using AE1 are in a range of 3.7 to 5.2 percent with the coefficient of variation 10.0 percent and air contents using AE2 are in a range of 3.7 to 5.2 percent with the coefficient of

variation 9.8 percent, the air contents using AE3 are in a range of 2.5 to 7.0 percent with the coefficient of variation 35.2 percent, the air contents using AE4 are in a range of 2.7 to 6.4 percent with the coefficient of variation 25.0 percent. Moreover, when AE1 and AE2 are used, the fluctuations in air contents are extremely small and stable air contents are obtained with small dosages, even though the loss on ignition of the fly ashes varies. This contrasts with the results obtained when AE3 and AE4 are used, where the variations are much greater.

Table 5 on next page

Table 6

Kind of Fly Ash	AE Admixture		Time-dependent Change in Air Content (%)		
	Kind	Dosage ¹⁾ (%)	0 min.	30 min.	60min.
Example	F4	AE1	0.0350	5.0	4.8
		AE2	0.0290	5.0	4.9
Comparison Example		AE3	0.0075	4.3	3.0
		AE4	0.1200	5.5	5.0

Note ¹⁾ Dosage of AE admixture solids percent by weight of cement or total quantity of cement and fly ash.

Table 6 gives the results of testing time-dependent changes in air contents of concretes using AE1, AE2, AE3 and AE4. The air contents of concretes using AE1 and AE2 show hardly any decline even after elapse of a period of 60 minutes. However, in the case of AE3, compared with AE1 and AE2, although air can be entrained with a low dosage, the reduction in air content after elapse of 60 minutes is considerable. In the case of AE4, although reduction in air content after the elapse of 60 minutes is small, the dosage required is very high in comparison with AE1 and AE2.

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¹⁾ Dosage of AE admixture solids percent by weight of cement or total quantity of cement and fly ash.

¹⁾ Dosage of AE admixture solids percent by weight of cement or total quantity of cement and fly ash.

Claims:

1. An air-entraining admixture which comprises

- (a) a fatty acid-based surface active agent; and
- (b) a non-ionic surface active agent;

- 5 the fatty acid-based surface active agent (a) being selected from C₁₂₋₂₄ alkanolic acids and their alkali metal, lower alkylamine and lower alkanolamine salts, and the non-ionic surface active agent (b) being selected from materials of the formula



- 10 where Ph(R) represents a phenyl group substituted with R, R being C₈₋₉alkyl and n is from 1 - 50.

2. An air-entraining admixture according to claim 1, wherein the admixture additionally comprises (c) a salt selected from the group consisting of salts of alkyl sulphonates, alkylaryl sulphonates, sulphate esters of higher alcohols and resinsates.
- 15 3. An air-entraining admixture according to claim 1, wherein the fatty acid-based surface active agent (a) is selected from fatty acids, or salts thereof, preferably the salts of alkali metals or amines.
4. An air-entraining admixture according to claim 3, wherein the alkali metal salts are those of sodium and potassium, and the preferred salts of amines are preferably those of low molecular weight alkylamines and alkanolamines, preferably those of
- 20 triethylamine or triethanolamine.
5. An air-entraining admixture according to claim 1, wherein the R substituent is selected from octyl and nonyl groups.

6. An air-entraining admixture according to claim 1, wherein the number n of oxy-ethylene units per molecule is in the range of 1 to 50, preferably from 20 - 30 oxy-ethylene units per molecule.
25
7. An air-entraining admixture according to claim 2, wherein the additive (c) is an alkali metal salt, preferably of sodium and potassium, or triethanolamine salt.
8. An air-entraining admixture according to claim 1, wherein the weight ratios of the individual components are from 10 - 90% (based on active material) of surface-active agent (a), from 90 - 10% from non-ionic surface-active agent (b) and, when
30 component (c) is present, no more than 20% thereof.
9. A process of preparing an aerated, fly ash-containing cementitious composition using a fly ash which has a loss on ignition of more than 5%, comprising the addition to the cementitious composition including the fly ash of an air-entraining
35 admixture according to claim 1 or claim 2.
10. An aerated fly ash-containing cementitious composition in which the fly ash has a loss on ignition of more than 5%, the composition comprising an air-entraining admixture according to claim 1 or claim 2.



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Claims searched: 1 to 10

Examiner: Miss M. M. Kelman
Date of search: 21 September 1995

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:
UK CI (Ed.N): C1H HAP HCA HCB HCC HCE HXL HXX
Int CI (Ed.6): C04B 24/00, 24/02, 24/08, 24/24, 24/28, 24/32
Other: ONLINE:CHEMENG, PATENTS

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X,Y	GB 0787187 A	I.C.I see page 1, lines 41 to 83, and Examples 2 and 4	X:1,3,4,5, 6,8 Y:9,10
X,Y	EP 0359068 A2	MERBABU CORPORATION see claims 1,3,4,7,8,9 and 10, page 3, line 48 to page 4, line 56 and the Examples	X:1,3,4 Y:9,10
Y	EP 0342011 A1	UNILEVER see whole document	9,10
Y	US 4828619 A	FUJISAWA see the claims and column 2, lines 33 to 35	9,10

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
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